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भारतीय मानक

ब्रोमीन, तकनीकी — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

BROMINE, TECHNICAL — SPECIFICATION

(*Second Revision*)

UDC 661·46

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 1992 **Price Group 2**

Acids, Alkalies and Halides Sectional Committee, CHD 02

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Acids, Alkalies and Halides Sectional Committee had been approved by the Chemical Division Council.

Bromine is used in the manufacture of ethylene dibromide for antiknock fluids and bromide salts. It is also used in shrinkage-proof wool, as an intermediate for fumigants, as fire extinguisher fluid and military poison gas.

This standard was originally published in 1962 and subsequently revised in 1974 incorporating requirements for iodine content and sulphates alongwith their methods of test. In this revision, requirements for bromine and chlorine content have been modified keeping in line with the available manufacturing practices in the country. Also, the method of test for chlorine has been suitably modified. The requirement for relative density has been deleted.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BROMINE, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of sampling and test for bromine: technical.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard.

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
1260 (Part 1) : 1973	Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods *(first revision)*
4905 : 1968	Methods for random sampling
5296 : 1979	Chloroform, pure and technical (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of reddish-brown fuming liquid.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods given in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of the table.

Table 1 Requirements for Bromine, Technical

(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Bromine, percent by mass, *Min*	98·5	A-2
ii)	Chlorine (as Cl,) percent by mass, *Max*	0·5	A-3
iii)	Non-volatile matter. percent by mass, *Max*	0·05	A-4
iv)	Iodine (as I), percent by mass, Max	0·05	A-5
v)	Sulphates (as SO_4), percent by mass, *Max*	0·015	A-6

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in glass bottles with lead caps or ground glass stoppered bottles sealed with an inert material. The bottles shall then be packed in expanded mica, diatomaceous silica or other inert absorbents. For larger packings, homogeneously lead-lined mild steel drums or monel metal drums conforming to the rules made by the Government of India and in force shall be used.

4.2 Marking

The containers shall be securely closed and marked with the following information :

a) Name of the material;

b) Indication of the source of manufacture;

c) Net mass; and

d) Batch number or lot number in code or otherwise.

4.2.1 The containers shall prominently display the words CORROSIVE HANDLE WITH CARE, and the appropriate caution label [see IS 1260 (Part 1) : 1973].

5 SAMPLING AND CRITERIA FOR CONFORMITY

Representative samples of the material shall be drawn and judged for conformity to the requirements of this specification in accordance with the procedure prescribed in Annex B.

ANNEX A

(*Clause* 3.2 and *Table* 1)

ANALYSIS OF BROMINE, TECHNICAL

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF BROMINE

A-2.1 Method A (Referee Method)

A-2.1.1 *Reagents*

A-2.1.1.1 *Potassium iodide*

A-2.1.1.2 *Standard sodium thiosulphate solution* — 0·1 N. The solution shall be standardized before use.

A-2.1.1.3 *Starch indicator solution*

Triturate 5 g of starch and 0·01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for 3 minutes. Allow the solution to cool and decant off the supernatant clear liquid.

A-2.1.2 *Procedure*

Dissolve about 10 g of potassium iodide in 30 ml of water and transfer the solution to a clean 100-ml volumetric flask. Weigh the flask accurately. Add about 0·5 ml of the material to the flask, stopper immediately and weigh again. Dilute the contents of the flask to 100 ml and mix well. Pipette out 25 ml of the solution from the flask into a conical flask and titrate with standard sodium thiosulphate solution using freshly prepared starch indicator solution.

A-2.1.3 *Calculation*

$$\text{Bromine, percent by mass} = \frac{31{\cdot}97\, V N}{M}$$

where

V = volume, in ml, of standard sodium thiosulphate solution used in the titration;

N = normality of the standard sodium thiosulphate solution; and

M = mass in g of the material taken for the test.

A-2.1.3.1 In the value of bromine percentage as obtained in **A-2.1.3**, make a correction for the chlorine present by subtracting the percentage result for chlorine obtained in **A-3.3** multiplied by 2·25.

A-2.2 Method B (Routine Method)

The bromine content is calculated by subtracting the sum of impurities from 100.

$$\text{Bromine content, percent by mass} = 100 - (X_1 + X_2 + X_3 + X_4)$$

where

X_1 = chlorine content, percent by mass (*see* **A-3.3**);

X_2 = non-volatile matter, percent by mass (*see* **A-4.2**);

X_3 = iodine content, percent by mass (*see* **A-5.3**);

X_4 = sulphates, percent by mass (*see* **A-6.3**)

NOTE — The limits of impurities for iodine and sulphates shall be taken at the maximum permissible levels, for example, iodine as 0·05 percent by mass and sulphates as 0·015 percent by mass.

A-3 DETERMINATION OF CHLORINE

A-3.1 Reagents

A-3.1.1 *Zinc Filings*

A-3.1.2 *Concentrated Nitric Acid* — conforming to IS 264 : 1976.

A-3.1.3 *Standard Silver Nitrate Solution* — 0·1 N.

A-3.1.4 *Nitrobenzene*

A-3.1.5 *Standard Ammonium Thiocyanate Solution* — 0·1 N.

A-3.1.6 *Ferric Alum Indicator* — saturated solution.

A-3.2 Procedure

Add 50 ml of water to a 250-ml volumetric flask fitted with a stopper and weigh (M_1). To this add about 3 ml of liquid bromine and weigh again (M_2). The difference between M_2 and M_1 gives the exact amount (M) of the material taken for the test. Add about 0·5 g of zinc dust and shake thoroughly for 10 minutes. Heat the flask on a steam-bath to remove the excess of bromine. Dissolve the residue in 75 ml of water and 25 ml of concentrated nitric acid. Heat the resulting solution to boiling on a water bath for 5 minutes. Pass a gentle stream of air for 20 minutes through a tapered in-let tube extending to the bottom of the flask to completely remove bromine. Allow the flask to cool and add 20 ml of standard silver nitrate solution. To this add 10 drops of nitrobenzene and shake vigorously for one minute. Titrate the excess silver nitrate with standard sodium thiocyanate solution using ferric alum indicator, shaking well between successive additions of the titrant. Carry out a blank determination using the same quantities of all the reagents.

A-3.3 Calculation

$$\text{Chlorine (as Cl), percent by mass} = \frac{3\text{·}546\,(V_1 - V_2)\,N}{M}$$

where

V_1 = volume, in ml, of standard ammonium thiocyanate solution used in the blank determination;

V_2 = volume, in ml, of standard amonium thiocyanate solution used in test with the material;

N = normality of standard ammonium thiocyanate solution; and

M = mass in g of the material taken for the test.

A-4 DETERMINATION OF NON-VOLATILE MATTER

A-4.1 Procedure

Transfer exactly 5 ml of the material to a weighed silica dish. Volatilize the material on a steam-bath under hood and dry the dish for one hour at 105 ± 5°C. Cool in a desiccator and weigh again.

A-4.2 Calculation

$$\text{Non-volatile matter, percent by mass} = \frac{100 \times M}{V \times D}$$

where

M = mass in g of the residue;

V = volume in ml of the material taken for the test; and

D = relative density of the material.

A-5 TEST FOR IODINE

A-5.1 Apparatus

A-5.1.1 *Nessler Cylinders* — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 *Zinc Dust*

A-5.2.2 *Ferric Chloride Solution* — 10 percent (*m/v*).

Dissolve 10·0 g of ferric chloride in 100 ml of water and filter.

A-5.2.3 *Chloroform — see* IS 5296 : 1979.

A-5.2.4 *Standard Potassium Iodide Solution*

Dissolve 0·202 8 g of potassium iodide in water and dilute to 100 ml in a volumetrtic flask. One millilitre of this solution is equivalent to 0·001 55 g of iodine.

A-5.3 Procedure

Shake 1 ml of the material with 50 ml of water and 3 g of zinc dust until all the bromine is decolorized. Filter the contents into a Nessler cylinder. Add to the filtrate 1 ml of ferric chloride solution, 5 ml of chloroform and shake well. In another Nessler cylinder, take 1 ml of standard potassium iodide solution and to this add 50 ml of water, 1 ml of ferric chloride solution, 5 ml of chloroform and shake thoroughly. Compare the colour of the cylinders.

A-5.3.1 The material shall be taken as not having exceeded the limit prescribed in Table 1 if the intensity of pink or violet colour of the chloroform layer in the first cylinder is not deeper than that of the colour of the chloroform layer in the second cylinder. This colour intensity corresponds to 0·05 percent iodine in the sample.

A-6 TEST FOR SULPHATES

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 *Dilute Ammonium Hydroxide Solution* — 10 percent (*v/v*).

A-6.2.2 *Dilute Hydrochloric Acid* — 1 N.

A-6.2.3 *Barium Chloride Solution* — Approximately 10 percent *m/v*.

A-6.2.4 *Standard Sulphate Solution*

Dissolve 0·1814 g of potassium sulphate in water and make up the volume to 1 000 ml. One millilitre of the solution contains 0·1 mg of sulphate (as SO_4).

A-6.3 Procedure

Take 1·3 ml of the material in a 100-ml porcelain evaporating dish with 30 ml of water. Add slowly 15 ml of dilute ammonium hydroxide solution and evaporate to dryness. Dissolve the white residue obtained in 80 ml of dilute hydrochloric acid. Take 40 ml of this solution in a Nessler cylinder and add 1 ml of barium chloride solution; mix well and allow to stand for 1 hour. Similarly, in another Nessler cylinder, take 3 ml of standard sulphate solution, 40 ml of dilute hydrochloric acid and 1 ml of barium chloride solution; mix and set aside for 1 hour.

A-6.3.1 The material shall be taken as not having exceeded the limit prescribed in Table 1 if the turbidity produced with the material is not greater than that produced with the standard sample. This standard turbidity corresponds to 0·015 percent sulphate content in the material.

ANNEX B

(*Clause* 5)

SAMPLING OF BROMINE, TECHNICAL

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 In drawing samples, the following precautions and directions shall be observed.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 Samples shall be placed in suitable, clean and dry containers on which the material has no action.

B-1.4 Sample containers shall be of such a size that they are almost completely filled with the sample.

B-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling and the date of sampling.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number of containers (*n*) to be selected for sampling from the lot shall depend on the size of the lot (*N*) and shall be in accordance with Table 2.

B-2.3 These containers shall be selected at random from the lot and in order to ensure the randomness of sampling, random sampling procedures as given in IS 4905 : 1968 may be followed.

Table 2 Number of Containers to be Drawn for Sampling

(*Clause* B-2 2)

Lot Size (*N*) (1)	Number of Containers to be Selected (*n*) (2)
Up to 15	2
16 ,, 25	3
26 ,, 100	4
101 ,, 300	5
301 ,, 500	6
501 ,, 800	8
801 ,, 1 000	10

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

Take out not less than 50 ml of the material from each container selected for sampling, the total volume of the material collected from all the containers being not less than 500 ml. This composite sample shall be divided into three equal parts not less than 100 ml each, and each part shall be transferred to a clean dry amber glass bottle. The bottles shall be sealed air-tight and labelled with all the particulars of sampling.

B-3.2 One set of test samples shall be sent to the purchaser and one to the supplier. The third set bearing the seal of the purchaser and the supplier shall constitute the referee sample, to be used in case of dispute.

B-4 TEST

B-4.1 Tests for all the characteristics given in Table 1 shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to the specification if the test results on the composite sample meet the requirements given in Table 1.

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